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(71) Applicant
Sandoz Ltd.

(Incorporated in Switzerland)

35 Lichtstrasse, CH–4002 Basle, Switzerland

(72) Inventors
Hermann Gilgen
Wolfgang Groebke

(74) Agent and/or Address for Service
B. A. Yorke & Co.,
98 The Centre, Feltham, Middlesex TW13 4EP

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(54) Hydroxyphenyl benzotriazole UV light absorber aqueous dispersions and their use in dyeing processes

(57) Stable dispersions comprise

20 to 45% of an insoluble or sparingly soluble U.V. absorber of the 2-(2'-hydroxyphenyl)benztriazole series having an average particle size of less than 5 μm;

7 to 15% of a dispersing agent that is a substantially salt-free condensation product of sulphonated tolylether and formaldehyde; and

40 to 73% water;

the percentages being by weight of the total composition.

These compositions are suitable for applying U.V. absorbers of the benztriazole series to a substrate in the form of a dispersion in water. They may be added to dye baths containing disperse dyes to give products with good light fastness.

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SPECIFICATION

Improvem nts in or relating to organic compounds

The invention relates to compositions of water-insoluble or very sparingly soluble U.V. absorbers that are stable to storage.

The problem with water-insoluble or very sparingly soluble U.V absorbers, particularly of the 2-(2'-hydroxyphenyl)benztriazole series, is that they tend to be difficult to disperse in water. To date, it has not been possible to produce concentrates of such U.V. absorbers that are stable for any length of time. For example, it is possible to form an aqueous dispersion of such U.V. absorbers after 90 hours or more of milling. However, after a short time the dispersion breaks up forming a clear aqueous phase with practically no U.V. absorber and a gelatinous precipitate, which after a few days is so compact that it cannot be stirred or shaken to reform the dispersion.

To alleviate this problem there is provided according to the invention a composition comprising:

20 to 45% of an insoluble or sparingly soluble U.V. absorber of the 2-(2'-hydroxyphenyl)benztriazole series having an average particle size of less than 5 μm;
7 to 15% of a dispersing agent that is a substantially salt-free condensation product of sulphonated tolylether and formaldehyde; and
40 to 73% water;

the percentages being by weight of the total composition.

Compositions according to the invention may include one or more of the following:

(a) 0.5 to 1.5% of a solubilising agent
(b) 0.1 to 0.2% of a buffer
(c) 0.8 to 2% of a nonionic surfactant; and
(d) 0.1 to 0.3% of an antifouling agent.

Preferred dispersing agents are those commercially available, (for example Baykanol SL) as described in US Patent 4,386,037, especially Example 15. The contents and preferences of this Patent are incorporated herewith by reference. The preferred amount of formaldehyde used per mol of sulphonated tolylether is from 0.2 to 3 moles.

Preferred U.V. absorbers of the 2-(2'-hydroxyphenyl)benztriazole series are those described in Japanese Kokai 56–31084 and Swiss Patent 494,060. The Kokai and the Swiss Patent are incorporated herein by reference.

Preferably the average particle size of the U.V absorbers used in a composition according to the invention is less than 1 μm.

Preferred non-ionic surfactants are addition products of C_{1-12} alkylphenol (preferably nonylphenol) and ethylene oxide and/or propylene oxide. Preferably the ratio of phenol to alkylene oxide is 1:1 to 1:10.

Preferred solubilising agents are addition products of poly-C_{1-4}alkylene glycols (preferably polypropylene or polyethylene glycol) with propylene oxide and/or ethylene oxide. More preferably the solubilising agents are the addition products of polypropylene glycol and propylene oxide and ethylene oxide, most preferably having a molecular weight of the polyoxypropylene part of 1500 to 2500 and 40 to 60% polyoxyethylene units, particularly preferred being "Pluronic" type products.

Preferred antifouling agents are fungicides for example Giv Gard, a commercially available product.

Preferably the buffer is a phosphoric acid (e.g. orthophosphoric acid).

Preferred compositions according to the invention comprise

25 to 40% of 2-2-(2'-hydroxy-3'-tert.butyl-5'-methyl-phenyl)-5-chlorobenztriazole;
8 to 12% of salt-free condensation product of ditolylethersulphonate and formaldehyde;
0.8 to 1.2% of the addition product of polypropylen glycol, propyleneoxide and ethyleneoxide having a molecular weight of the polyoxypropylene part of 1700 to 2300; and 40 to 60% polyoxyethylene units in the polymer;
0.12 to 0.18% of a phosphoric acid; and
1.0 to 1.5% of the addition product of a C_{1-12}alkylphenol and a C_{2-3}alkyleneoxide; and
45 to 65% demineralised water;

the percentages being by weight of the total composition.

Preferably the pH of a composition according to the invention is between 4.5 and 5.8, more preferably 5.0 to 5.5.

Compositions according to the invention are useful for use in dyeing, padding or printing processes preferably in a concentration of 0.5 to 2.0% based on the weight of substrate to be treated.

Compositions according to the invention have excellent stability to storage.

Compositions according to the invention are also suitable as aftertreatment compositions being applied by padding, printing or exhaustion techniques as a 0.5 to 2.0% dispersion in water.

Further, according to the invention there is provided a process for dyeing a substrate comprising applying to the substrate

0.1 to 5% of a dye (preferably a disperse dye)
0.1 to 5% of a composition according to the invention

[the percentages being by weight of the substrate being dyed]
preferably in an aqueous medium in a liquor to goods ratio of 10:1 to 60:1.

Substrates dyed with disperse dyes and a U.V. absorber according to the invention show excellent light fastness properties.

The average particle size can be measured by placing a sample on a microscope on which a scale has been superimposed.

The invention will now be illustrated by the following Examples in which all parts and percentages are by weight and all temperatures are in °C.

Example 1

The following are mixed together in a vessel

40 parts of 2-(2'-hydroxy-3'-tert.butyl-5'-methyl phenyl)-5-chlorobenztriazole

10 parts of a commercially available condensation product (salt-free) of ditolylether sulphonate and formaldehyde (Baykanol SL);

0.25 parts of a fungicide (Giv Gard)

1 part of a commercially available product of 9 moles ethyleneoxide and 1 mol nonylphenol; and

1 part of commercially available Pluronic P 75 (from BASF/Wyandotte Corp.) [the addition product of propylene glycol and propylene oxide followed by the further reaction with ethylene oxide; the product having a molecular weight for the polyoxypropylene part of 2050 (approx.) and having about 50% polyoxyethylene units in the polymer] in 40 parts of demineralised water.

To this mixture is then added about 0.15 parts of ortho phosphoric acid to bring the pH to 5 to 5.5. The mixture is then milled in a bead mill with silicoquarzite beads until the average size of the particles of the U.V. absorber is less than 1 um, which occurs after about 30 minutes. The resulting dispersion is filtered from the silicoquarzite beads, the beads are washed with 7.6 parts of water and the filtrate and water with which the beads have been washed are added together.

The resulting concentrate is stable as a dispersion for a number of months even at temperatures above 50°C.

Examples 2 and 3

Example 1 is repeated using 40 parts of
2-(2'-hydroxy-5'-methylphenyl)benztriazole (Example 2) and 40 parts of 2-(2'-hydroxy-3',5'-di-tert.-butyl)benztriazole instead of 40 parts of
2-(2'-hydroxy-3'-tert.butyl-5'-methylphenyl)-5-chlorobenztriazole.

Application Example

A dyebath is made up of the following:

1.5 parts of C.I. Disperse Red 74
1.5 parts of the dispersion of Example 1; and
197 parts of demineralised water.

The pH is brought to 5 by the addition of ammonium sulphate/formic acid in a ratio of 10:1.

This bath is warmed to 60° in a dyeing autoclave and 100 parts of a polyester fabric (of sufficient quality for use in automobile upholstery) is then added, the autoclave is closed and the circulating bath is heated to 135°. Dyeing is carried out at 135° for 20 minutes after which the bath is cooled to 80° and the dyed substrate is removed from the bath. It is then washed, reductively cleared, washed again and dried. The light fastness of the dyed fabric is substantially better than that dyed without the benztriazole present.

Instead of usig 1.5 parts of the dispersion of Example 1, Application Example can be repeated

using 1.5 parts of the dispersion of Example 2 or 3.

CLAIMS

1. A composition comprising

20 to 45% of an insoluble or sparingly soluble U.V. absorber of the 2-(2'-hydroxyphenyl)benztriazole series having an average particle size of less than 5 μm;
7 to 15% of a dispersing agent that is a substantially salt-free condensation product of sulphonated tolylether and formaldehyde; and
40 to 73% water;

the percentages being by weight of the total composition.

2. A composition according to Claim 1 comprising additionally one or more of the following:–

(a) 0.5 to 1.5% of a solubilising agent
(b) 0.1 to 0.2% of a buffer
(c) 0.8 to 2% of a nonionic surfactant; and
(d) 0.1 to 0.3% of an antifouling agent.

3. A composition according to Claim 1 or Claim 2 in which the particle size of the U.V. absorber is less than 1 μm.

4. A composition according to Claim 1 comprising 25 to 40%

25 to 40% of 2-(2'-hydroxy-3'-tert.butyl-5'-methyl-phenyl)-5-chlorobenztriazole;
8 to 12% of a salt-free condensation product of ditolylethersulphonate and formaldehyde;
0.8 to 1.2% of the addition product of polypropylene glycol, propyleneoxide and ethyleneoxide having a molecular weight of the polyoxypropylene part of 1700 to 2300; and 40 to 60% polyoxyethylene units in the polymer;
0.12 to 0.18% of a phosphoric acid; and
1.0 to 1.5% of the addition product of a C_{1-12}alkylphenol and a C_{2-3}alkyleneoxide; and
45 to 65% demineralised water;

the percentages being by weight of the total composition.

5. A composition substantially as herein described with reference to any one of Examples 1 to 3.

6. A process for dyeing a substrate comprising applying to that substrate

0.1 to 5% of a dye and
0.1 to 5% of a composition according to Claim 1, the percentages being based on the substrate to be dyed.

7. A substrate to which a composition according to any one of Claims 1 to 5 has been applied.

8. A process substantially as herein described with reference to the Application Example.

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